Pumps and Holders

8

CHAPTER PREVIEW

In the past three chapters we've described the sources, lenses, and detectors that make up a TEM. The only other parts of the instrument you need to know about in detail are the parts that, if you are not careful, can seriously degrade the quality of the information you generate. These two parts are the holder in which you put your specimen and the vacuum that surrounds it. While there isn't much you can do to improve the vacuum, beyond buying a better microscope, there is a lot you can do that will degrade the quality of the vacuum in the column and, in doing so, contaminate your specimen. So we'll tell you a few basics about how the vacuum pumps work, and how the vacuum system is put together. Although the vacuum system is under computer control in most TEMs, you still affect the vacuum by what you put in the microscope. Consequently, you need to know what not to do on those occasions that you can degrade the vacuum.

The vacuum in the stage of a typical TEM is ~10^{-5} Pa, which compares with atmospheric pressure of ~10^{5} Pa. It is quite remarkable that we can transfer a specimen into the TEM, reducing the ambient pressure at its surface by 10 orders of magnitude in a matter of a few seconds. This rapid transfer is a testament to the skills of TEM designers, and particularly the construction of the specimen holder and the airlock system. Specimen holders are the physical contact between you and your specimen across this extraordinary vacuum range. Through the holder you have to transmit all the experimental variables that you want to inflict on your

D. B. Williams et al., *Transmission Electron Microscopy*

specimen. The most basic requirement is that you should be able to move the specimen laterally to look at different areas. In addition we'll describe how you can tilt, rotate, heat, cool, strain, and bias the materials that you are studying. In addition to transferring useful external variables to the specimen, the holder also transmits vibrations, drift, and contamination to the specimen and may be a source of X-rays that can confuse any microanalysis that you want to perform. Care of your specimen holders is extremely important since damaged or worn holders reduce the quality of the data generated by the microscope. If you are not careful, a $10,000 holder can easily limit the information generated by a million dollar TEM.

Pumps and Holders

8

8.1. VACUUMS

You know already that electrons are strongly scattered by atoms, which accounts for the versatility of TEM, and the need for thin specimens. Strong scattering also occurs in gases and we can't send coherent, controlled electron beams through the atmosphere, so all EMs operate under vacuum. This means that your specimen has to go through an airlock into the TEM. Therefore, you can only control your specimen remotely, not directly, and this makes TEMs more expensive to build. In addition to permitting the electron beam to travel through the instrument undisturbed, the vacuum also plays a role in keeping the specimen clean (or dirty). Contamination of the specimen by vacuum-borne contaminants such as hydrocarbons and water vapor can be a problem in many aspects of TEM. Generally, the better the vacuum, the less contamination, but it is the partial pressure of contaminants, not the absolute pressure, which is important. Fortunately, the vacuum systems in most TEMs today are reasonably clean, fully automated, and their operation is transparent. Despite this, you should have some understanding of vacuums and how to control them, so this chapter will cover, very superficially, the principles of vacuum systems and pumps. For a full exposition of vacuum technology for TEMs, read Bigelow (1995) or the equally informative user's guide by O'Hanlon (1980).

First of all, a word on units—which as usual are in disarray. The SI unit of pressure is the pascal (Pa); other non-SI units are the torr and the bar. You'll come across all three units in TEM texts and in manufacturers' handbooks, so you need to know the conversions. One bar is atmospheric pressure (~760 Torr) and is equivalent to ~10^5 Pa and so

One Torr is ~ 130 Pa, 1 Pa is 7.5×10^{-3} Torr.

We'll mainly use Pa, but since Torr is still very common terminology, we'll occasionally put highly approximate torr values in parentheses to remind you of the conversion. Since we deal with very low pressures, the numbers are small, although we perversely use the expression "high vacuum" for these low pressures. We can divide vacuums into rough, low, high, and ultrahigh. Pressures between 100 and 0.1 Pa (~1 and 10^{-3} Torr) are rough vacuums, 0.1–10^{-4} Pa (~10^{-3}–10^{-6} Torr) are low, 10^{-4}–10^{-7} Pa (~10^{-6}–10^{-9} Torr) are high vacuums (HV). If the pressure is <10^{-7} Pa (~10^{-9} Torr) you have an ultrahigh vacuum (UHV). These are approximate, not standardized definitions. A typical modern TEM has a pressure inside the column of ~10^{-7} Torr (1.3×10^{-5} Pa), which is in the HV range. UHV TEMs operate below 10^{-7} Pa and the gun region of an FEG TEM operates at ~10^{-9} Pa (10^{-11} Torr). To get an electron beam inside the TEM that is not scattered by the air molecules in the column, the pressure has to be < ~0.1 Pa and this was achievable with simple mechanical pumps in the early days of the instrument. But there are good reasons to operate at much lower pressures (higher vacuums), for which you need more sophisticated and more expensive apparatus.

Generally, we use one type of pump to create a rough vacuum and another type to create the higher vacuum. The TEM is kept permanently under vacuum, unless under repair or service. If you need access to the inside of the column to change specimens, electron sources, or photographic plates, you do this via an airlock system, which can be pumped separately, as we'll explain later. There are many different kinds of pumps used in TEMs, and you often have a choice when purchasing an instrument. As with most things, you get what you pay for; a clean UHV system is very expensive. We can divide pumps into roughing pumps and HV/UHV pumps, as we'll now discuss.

8.2. ROUGHING PUMPS

The most common roughing pump is a mechanical (rotary) pump in which a belt-driven, eccentrically mounted reciprocating mechanism sucks air through an inlet valve into a

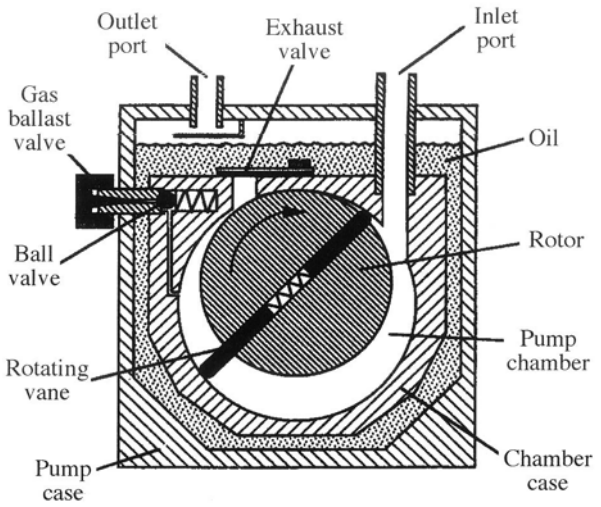

Figure 8.1. A mechanical pump for roughing vacuums. The eccentric motion of the pump creates a vacuum in the RH side when it rotates and the vacuum sucks air into the inlet valve. As the cylinder rotates further, it cuts off the inlet and forces the air through the outlet on the LH side, creating a vacuum again on the inlet side as it does so. Because of the constant contact between the rotating cylinder and the inside of the pump, oil is needed to reduce frictional heating.

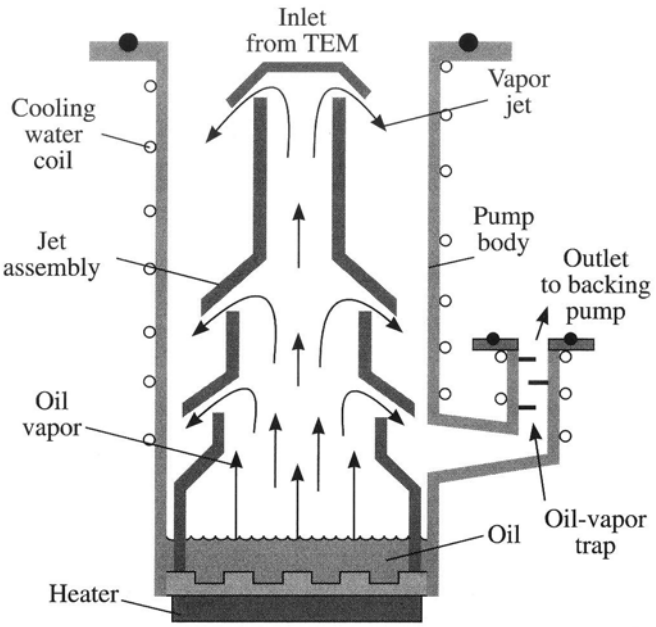

Figure 8.2. Principles of diffusion pump operation. A heater plate at the base of the pump boils synthetic oil. The expansion of the oil vapor on boiling creates a pressure, which forces the vapor up the central column and out of several holes. The stream of oil vapor pulls gas molecules out of the top of the pump down to the base, where the oil condenses and the air is pumped out of the base by a mechanical backing pump.

chamber and expels it through an exit valve, as shown in Figure 8.1. Such pumps are very reliable, relatively inexpensive, noisy and dirty, and only lower the pressure to ~10^{-1} Pa (~10^{-3} Torr). Mechanical pumps should be housed outside your TEM room, and connected to the column through a line that doesn't transmit their vibration. These pumps use a hydrocarbon oil as a medium. If you have such a pump, the line from the pump to the vacuum should contain a foreline trap to condense out oil vapor before it is deposited in the column. Also, the exhaust line from the pump must be well trapped to prevent (possibly carcinogenic) oil vapor escaping into the room where you are working. There are alternative "dry" roughing pumps which do not use oil. These are more expensive and somewhat less reliable; they do not pump to such low pressure.

8.3. HIGH/ULTRA-HIGH VACUUM PUMPS

8.3.A. Diffusion Pumps

These pumps use a hot plate to boil oil, which then forms a series of concentric vapor jets. The jets drag air molecules out of the microscope as shown in Figure 8.2, then condense onto a cold surface, freeing the air molecules which are extracted by the mechanical pump "backing" the diffusion pump. While this may seem an inefficient way to move air, diffusion pumps can in fact transport hundreds of liters of air per second, which is quite sufficient to pump out a TEM column. With no moving parts, diffusion pumps are inexpensive and very reliable, but they need external water cooling to aid condensation of the vapor. Failure of the water cooling and burn-out of the hot plate are about the only possible causes of failure. The absence of moving parts ensures vibration-free operation. As with the mechanical pump, the oil diffusion pump would contaminate the vacuum in the TEM if oil vapor were to escape into the column. To minimize this you must use synthetic nonhydrocarbon oils with low vapor pressures, such as Fomblin™ or Santovac™. A liquid-N_2 cold trap sits on top of the pump and condenses out any residual oil molecules. If you have diffusion pumps you must keep the cold traps full of liquid N_2 to maintain a clean system.

Diffusion pumps are capable of very efficient pumping from ~10^{-1} to ~10^{-9} Pa (10^{-11} Torr) and, if properly trapped, will provide a clean UHV system that is very reliable. The VG series UHV DSTEMs use only oil diffusion pumps to attain UHV conditions.

8.3.B. Turbomolecular Pumps

Turbomolecular pumps, or turbopumps, as the name implies, use a turbine to force gases from the microscope.

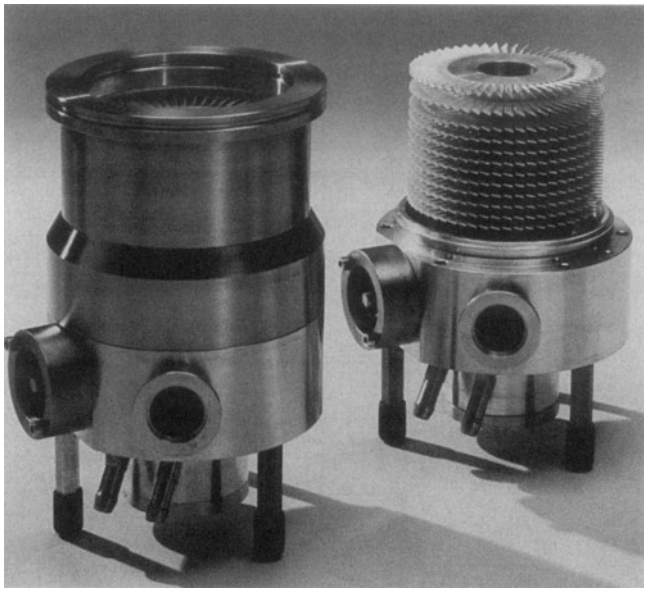

Figure 8.3. A turbopump (with and without its casing) which is nothing more than a small turbine that rotates at high speed. Like a jet turbine it pulls air in at the front end and forces it out of the back. The blades are designed like airfoils to enhance the flow of gas through the system.

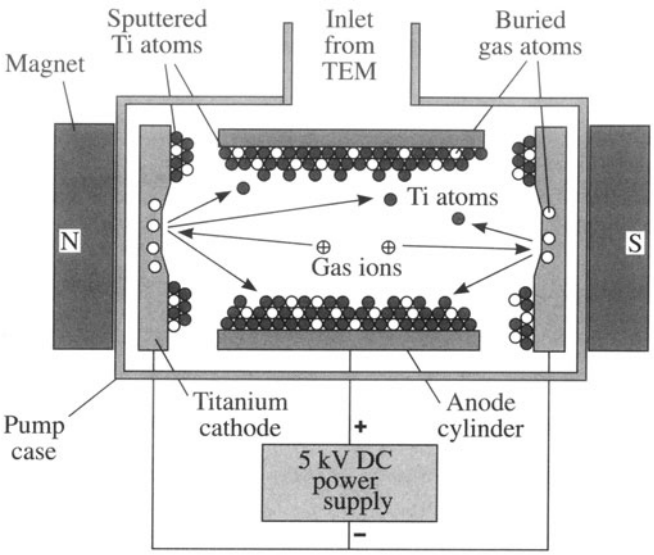

Figure 8.4. Schematic diagram showing how ion pumps trap ionized gas atoms by layers of Ti atoms at electrodes. Once trapped, the ions cannot escape until the pump is turned off.

They have many parts moving at high speeds (in excess of 20,000–50,000 rpm is common), so they are more liable to fail than diffusion pumps. The mechanics of the pump are very simple, as you can appreciate from Figure 8.3. They do not use oil so they don't introduce hydrocarbons to contaminate the microscope, and the best models (unlike earlier versions) are very quiet and almost vibration-free. In fact, modern turbopumps are being used to prepump the specimen chamber when this is critical, as in the cryotransfer technique (see Section 8.10). If you buy a turbopump, make sure to specify that its use will not transmit vibrations to the TEM column, where they would destroy the image resolution. The turbopump can start (slowly) at ambient pressures, increasing speed as the pressure is lowered, ultimately providing UHV conditions at high enough speeds. It is usual, however, to back the turbopump with a dry mechanical pump.

Mechanical, diffusion, and turbopumps are all exhaust pumps; they pull in air from one end and expel it from the other.

8.3.C. Ion Pumps

Ion pumps do not contain oil, so they cannot contaminate the TEM column. They also have no moving parts, relying solely on the ionization process to remove air. The ion pump emits electrons from a cathode, and these spiral in a magnetic field (see Section 6.3) and ionize air molecules, which are then attracted to the cathode. The energetic gas ions sputter Ti atoms from the cathode and they condense throughout the system, mainly on the cylindrical anode, trapping gas atoms. Thus ion pumps remove gas atoms in two ways; by chemisorption on the anode surfaces and by electrical attraction to the cathodes. The smaller the ion current between the electrodes, the lower the vacuum, so the pump acts as its own vacuum gauge. Ion pumps are only efficient at high vacuums, so they are usually switched on after a diffusion pump has lowered the pressure to $< \sim 10^{-3}$ Pa (10^{-5} Torr). It is common to add ion pumps directly to the stage or gun chambers of TEMs to focus their pumping action on these important regions. Since these pumps are very common on TEMs, we include a diagram (Figure 8.4) showing how they operate.

8.3.D. Cryogenic (Adsorption) Pumps

As the name implies, cryogenic pumps (cryopumps) rely on liquid N_2 to cool molecular sieves with large surface areas. The cold surface efficiently removes air molecules from ambient pressure down to $\sim 10^{-4}$ Pa (10^{-6} Torr). Because they are oil-free, cryopumps are also used to back out ion pumps and prevent their accidental contamination through backstreaming from oil-bearing pumps.

We also use cold surfaces to enhance vacuums in the stage of most non-UHV TEMs. Such "cold fingers" or "anticontaminators" provide an alternative site (to your specimen) for condensation of residual components in the vacuum.

Ion pumps and cryopumps are trapping pumps. They keep the air molecules within them and release them when turned off or warmed up, respectively.

The same is true if the anticontaminator in your stage is allowed to warm up; then it will degrade the vacuum around your specimen. So you must use another pump, such as a diffusion or mechanical pump, to remove the air molecules as they are released from captivity. Otherwise, this outgassing will degrade the quality of the vacuum around your specimen, increasing contamination.

8.4. THE WHOLE SYSTEM

As shown schematically in Figure 8.5 the TEM has separate pumping systems: one that evacuates the column and one that pumps the camera and screen chamber. We pump the camera separately because the film is one of the primary causes of vacuum degradation since outgassing occurs from the emulsion that contains the AgI grains. So this part of the TEM is usually pumped by a combination mechanical/diffusion pump. The stage is often pumped by a separate ion pump, turbopump, or cryopump, or some combination of these. If the instrument has an FEG, then there is a separate UHV pumping system for the gun region, which often consists of several ion pumps. Each part of the vacuum system consists of roughing pumps (mechanical or turbo) that pump out the appropriate part of the microscope to a pressure below which the HV/UHV pumps can operate.

Looking at Figure 8.5, there are three valves, which are now all computer-controlled:

- #1 connects the mechanical pump to the column (the roughing valve).
- #2 connects the mechanical pump to the bottom of the diffusion pump (the backing valve).
- #3 connects the diffusion pump directly to the TEM column (the butterfly valve).

If you're pumping down from atmospheric pressure, you first use the mechanical pump to back out the diffusion pump, till it gets to a low enough pressure so its heater can be safely switched on without oxidizing. So close #1, open #2, and close #3.

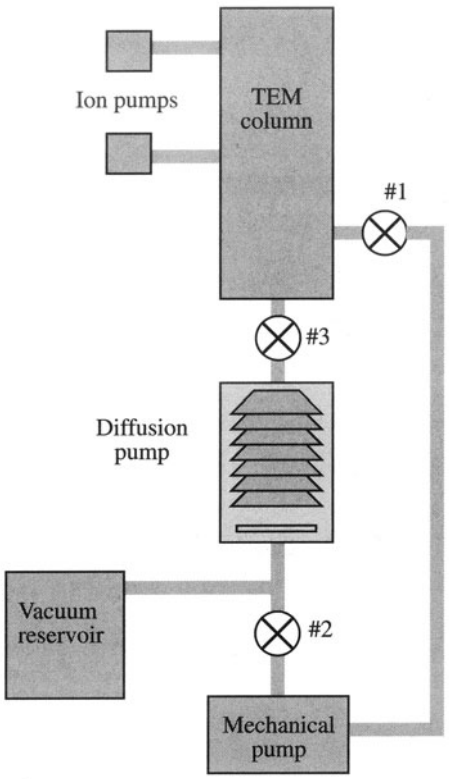

Figure 8.5. The principles of the TEM vacuum system. Often, the console display on the TEM will show a similar diagram. The mechanical pump can pump the column directly or back out the diffusion pump, which is connected directly to the base of the microscope. Ion pumps are often interfaced directly to the stage and gun areas. Computer-controlled valves separate the pumps from the column and from each other.

When the diffusion pump is warmed up, you rough out the column: open #1, close #2 and #3, until the column is at a low enough pressure that the diffusion pump can be used.

At this point, close #1, open #2 and then #3, so the diffusion pump is open to the TEM and also continuously backed by the mechanical pump. Alternatively, a vacuum reservoir is attached between the mechanical and diffusion pumps. When the reservoir is pumped to < 0.1 Pa, the mechanical pump is closed off and the diffusion pump exhausts into the reservoir. When the pressure builds in the reservoir, the mechanical pump will automatically switch on and lower the pressure.

Similar arrangements work for other pumps; e.g., the diffusion pump is used to lower the pressure in the stage and gun sufficiently for the ion pumps to be switched on, and so on. In most TEMs the stage and gun have sig-

nificantly better vacuums than the camera region, so the camera/screen is isolated from the rest of the column by a differential pumping aperture (not shown in Figure 8.5). This aperture often coincides with the BFP of the projector lens, since all the electrons have got to pass through it and the diffraction pattern in the BFP localizes all the electron trajectories close to the optic axis. A similar arrangement exists between the stage and gun in FEG systems to preserve the tip in case of a vacuum leak in the stage.

The advent of high-quality digital recording which will remove the need for film in the camera will do more to improve the quality of vacuums in TEMs than any advances in pumping technology.

8.5. LEAK DETECTION

Nature abhors a vacuum, as Francois Rabelais said in 1534, and that's the reason why the pumps have to keep pumping: the TEM leaks. But some leaks are too large for the pumps to handle, and then the instrument performance degrades. For example, you might not be able to run the electron gun, so your TEM is useless. Under these circumstances, you have to find the leak, cure it, and repump out the instrument (this is usually a job for your service engineer). Leak detection involves using a mass spectrometer, which can be put into the pumping lines of the microscope. You then release helium gas close to the various parts of the TEM where you suspect a leak (e.g., the stage airlock, which sees a lot of use, is a common point of failure). The small helium atoms are relatively easily sucked into the column through any leak and register on the mass spectrometer. When a leak is isolated, the TEM may have to be opened to the atmosphere to permit replacement of the defective part, such as the O-ring seals.

The most common cause of a leak is your specimen holder. The O-ring seal on the shaft of a side-entry holder (see the second half of this chapter) is easily contaminated with dust or a hair since it is continually inserted and extracted from the column, and left on the bench while you pore over it. Never touch the O-ring, make sure it doesn't dry out, but if it does, lubricate it with a very thin film of vacuum grease.

After repairing a leak, when you've pumped down again, it is often useful to "bake" the column. Baking means heating the internal surfaces to >100°C (or >150–200°C in UHV TEMs) to boil off residual water vapor and hydrocarbons that may have entered the system when it was down to air. Usually, you can bake by leaving the lenses running without their cooling water (check this *very* carefully with the manufacturer before proceeding). In some cases, special heating panels are constructed around the column. Baking can also introduce other leaks as the whole system expands and then contracts, so sometimes leak detection and cure is an iterative process. For UHV systems, you *must* bake to reach the ultimate vacuum, and the higher the temperature the better.

Be wary, however, since sometimes the TEM accessories, such as XEDS and EELS systems, are not designed to be baked to the same high temperature as the column.

8.6. CONTAMINATION: HYDROCARBONS AND WATER VAPOR

As we said right at the start of the chapter, the vacuum can be a source of contamination, particularly residual hydrocarbons from the pump oil which crack under the electron beam. Carbonaceous material then deposits on your carefully thinned specimen, making it difficult to do sensible high-resolution imaging or microanalysis. So a clean vacuum (one in which the hydrocarbon partial pressure is $< 10^{-9}$ Pa) is essential. Fortunately, most modern TEMs are relatively contamination-free, particularly if you use appropriate traps on the pumps and synthetic oils.

However, even if you've paid dearly for a clean vacuum system, contamination often occurs and it comes primarily through the airlock with your specimen. You can minimize this by heating the specimen to >100°C in a heating holder or with a halogen lamp in the prepump chamber, or cooling the specimen to liquid-N_2 temperatures in a cooling holder. It may help if the prepump chamber is pumped with an oil-free pump. More recently, plasma ashing of the specimen holder and specimen prior to insertion in the TEM has proven a very successful way to ensure a clean specimen, but this is expensive. Polymers and biological specimens can easily introduce hydrocarbon contaminants, as they outgas in the vacuum, so it is sensible to cool such specimens (since heating or plasma ashing destroys them). However, when you cool your specimen, it attracts water vapor which condenses as ice on the surface; so load your specimen first, then cool it down in the TEM before you switch on the beam. A low partial pressure of H_2O in the vacuum is obviously essential. Also, warm up any cooled specimens in the TEM before bringing them out to ambient atmosphere, otherwise they will immediately ice up (unless it's a *very* dry winter's day in Minnesota).

There will be more about this in the ensuing sections on specimen holders.

In addition to the specimen, you personally can be a major source of contamination unless you take care never to touch anything that will enter the vacuum, i.e., the specimen itself, the grids, specimen holder (beyond the O-ring seal on the rod), clamping rings, replacement diaphragms, new filaments, replacement Wehnelt, components of XEDS and EELS systems, etc. Use latex gloves whenever you load a specimen, and don't breathe on it. Store specimen holders and specimens in a dry box containing a desiccant such as silica gel, which should be replaced regularly. *Always* prepump fresh film in a vacuum desiccator (which is sometimes integrated into the TEM itself). Simple precautions like this will minimize contamination of your specimens and the microscope in general and bring a much greater return in terms of good data per TEM session.

8.7. SPECIMEN HOLDERS AND STAGES

To look at your specimen, you place it in a specimen holder and insert this assembly into the TEM stage. Therefore, there are two key components which are often not separated, namely, the holder and the stage. In this part of the chapter, we will emphasize the holder but the stage is also critical. Suitable design of the stage is the essential precursor to computer-controlled TEM, which is already appearing.

The cold trap, cold finger, or cryoblades are a critical part of the stage. Ideally, this cold finger will completely surround the specimen. However, the cold surfaces, usually brass, provide a source of stray electrons and X-rays which is undesirable for AEM (see Chapter 33), so these blades should be removable.

X-ray diffractometers use goniometers to hold and tilt the specimen; so do TEMs. Conventional SEMs use a stub on which you mount the specimen so that you can bring the specimen close to the objective lens. However, the new high-resolution SEMs use a specimen holder which is very similar to those used in the TEM, because the specimen is inserted inside the lens, rather than underneath and outside it.

The reason the specimen holder is so important in TEM is that your specimen must invariably be located within the objective lens and the aberrations associated with the objective lens determine the resolution of the TEM.

Historically, microscopists have used two different designs and a lot of what you'll read has a strong historical background.

- The traditional side-entry holder is a rod with a motor attached to tilt and/or rotate the specimen and a lead connecting it to a power supply and control box, or liquid-N_2 dewar.
- The traditional top-entry holder is a cartridge which you load into the TEM but is detached from the outside world when you use the microscope.

The actual cup that holds your specimen is either 2.3 mm or 3.05 mm in diameter, so the specimen disk or support grid has to be the same dimension, as we'll see in Chapter 10. The reasons for these dimensions are again partly historical. In the top-entry holder the specimen and part of the holder fit through the bore of the upper pole-piece (see Figures 6.7 and 6.8). Clearly, the specimen must be smaller than the bore diameter. So the original top-entry holders used small specimens.

Side-entry holders are more versatile and larger specimen dimensions first appeared when they were introduced. However, side-entry holders connect the specimen directly to the outside world via a long lever arm, which is undesirable, unstable, and also not necessary in many cases! Ideally, the side-entry holder should leave the specimen in the stage, not connected to the outside world, and all manipulations should be conducted through the stage itself, not the holder. This ideal is being approached as stages become more computer-controlled.

8.8. SIDE-ENTRY HOLDERS

Side-entry holders are now the standard, although their design has changed quite radically. The traditional design is shown in Figure 8.6. The key parts of the holder are:

- *The O-ring,* which is one mechanical link to the microscope column. Some holders have two O-rings and the gap between the O-rings is pumped separately to improve the vacuum.
- *The jewel bearing,* which is the other mechanical link to the microscope column. You push on this bearing to move your specimen back and forth and from side to side. Like the O-ring, you must keep the bearing clean otherwise the specimen will not be stable.
- *The cup,* which actually holds your specimen and thus provides the immediate environment which is seen by stray electrons and any X-rays coming down the column. So cups in holders for AEM are made of Be to minimize the gener-

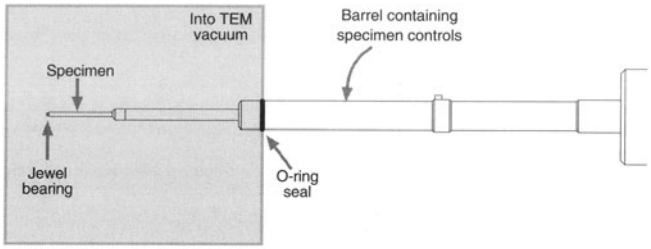

Figure 8.6. Principal parts of a side-entry holder that is held in the goniometer stage. The specimen is clamped into the cup at the end of the rod. A small jewel at the end of the rod (usually sapphire) fits into another jewel bearing in the stage to provide a stable base for manipulating the specimen. The O-ring seals the end of the holder inside the vacuum. Manipulating the specmen is accomplished from outside the column via controls within the rod.

ation of X-rays that would interfere with microanalysis.

- *The clamping ring or screw,* which holds the specimen in the cup. This ring, which may also be Be, must be carefully designed. It must hold your specimen firmly (so, e.g., magnetic disks cannot be pulled out of the cup by the lens field). However, the ring must not be so difficult to tighten that you put undue pressure on your specimen, since brittle disks may break as you are loading them. There are two kinds: screw-thread rings, which are easier to control and do not damage metals, but you'll find they may break ceramics because they transfer shear stresses to the disk; spring clips are difficult for the novice to master, but with practice you'll find they offer more control over the load that you put on the specimen, so we recommend them for the experienced ceramist. Unfortunately, no one makes Be spring clips!

8.9. TOP-ENTRY HOLDERS

Top-entry holders are becoming less common because they essentially preclude XEDS analysis in the TEM. Also, it is more difficult to design such holders so that the specimen can be manipulated (e.g., rotated or strained). Their great advantage was that they were much less susceptible to drift since they were not connected directly to the outside, so early HRTEM required top-entry holders. Today, however, all TEMS up to 400 kV use side-entry holders.

Another drawback of such holders is that the bore of the objective lens must be asymmetric, which actually limits the ultimate resolution by constraining the lens designer. Figure 8.7 shows a schematic diagram of such a holder.

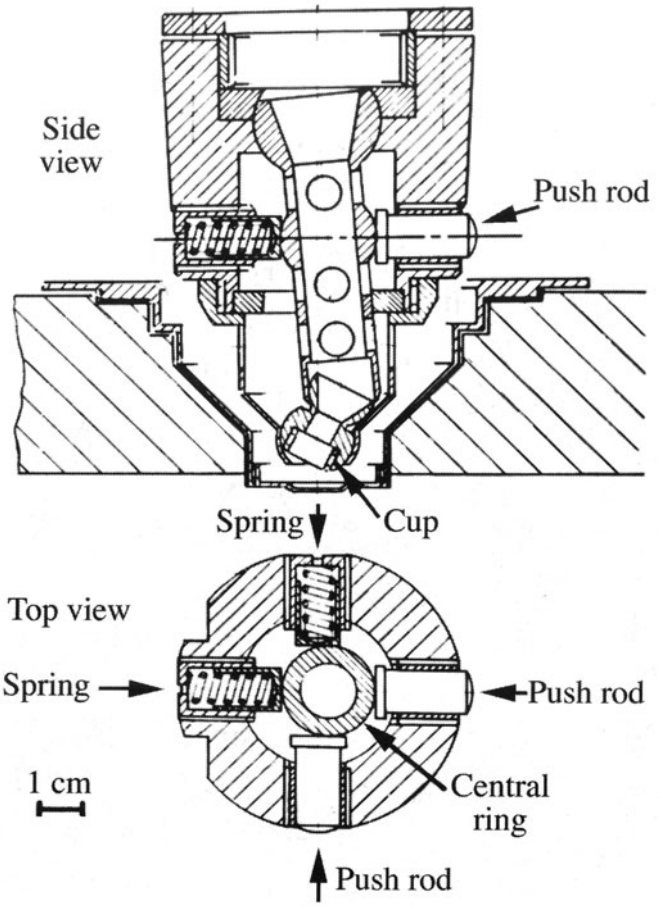

Figure 8.7. Cross section of a top-entry holder. The cartridge has a cone shape which fits into the tapered bore of the objective lens polepiece. The specimen sits in a cup at the base of a column through the cone down which the incident beam travels. Simple manipulations such as tilting or rotating require complex micromechanical design, since the specimen is at the base of the cartridge and completely surrounded by the polepiece. To tilt, e.g., as shown in the upper diagram, push rods are pressed against springs in two orthogonal directions, displacing a ring around the column (lower diagram), thus tilting the specimen cup.

8.10. DIFFERENT TYPES OF HOLDERS

One feature of TEMs which may surprise you if you are a new user is the wide variety of holders which is available. Figure 8.8 shows illustrations of some different designs for the side-entry holder:

- *Single-tilt holder:* This is the basic holder with which any novice should start practicing. You can only tilt around the axis of the rod. It is relatively cheap, robust, and can at least give you some idea of the usefulness of tilting a specimen for diffraction contrast studies.

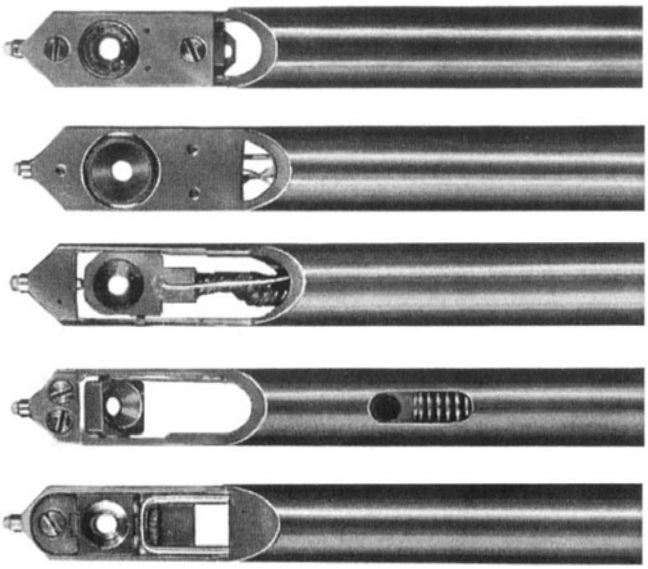

Figure 8.8. Examples of different designs for the side-entry holder. From the top, they are: a rotation holder, a heating holder, a cooling holder, a double-tilt holder, and a single-tilt holder.

- *Quick change holder:* This is also a single-tilt holder that clamps the specimen with a lever arm which you raise and lower onto your disk or grid. It doesn't put a high stress on the specimen, but it doesn't hold it very strongly either. Don't use it for magnetic specimens, but it's great for ceramics. Different retainers can be substituted for the clamp shown in Figure 8.8 (bottom), creating a more versatile multipurpose holder.
- *Multiple-specimen holder:* This is usually a single-tilt holder, but you can load up to five specimens into the column at one time, as shown in Figure 8.9A. A two-specimen, double-tilt version is also available (Figure 8.9B). Such holders can be useful if you are not very good at specimen preparation, or you want to compare different specimens under identical conditions without turning off the beam. However, in modern TEMs, specimen exchange is relatively quick, except in UHV instruments where the multiholder is probably more valuable.
- *Bulk specimen holder:* This holder is used for surface imaging and diffraction, e.g., using SE or BSE in a STEM or for reflection diffraction and imaging in a TEM (see Chapter 31 for more about these techniques). The bulk specimen is larger than the traditional 3-mm disk (usually ~10 mm x 5 mm) so if you can create a thin specimen of these dimensions, the bulk holder will allow you to sample more of your material at one time (Figure 8.10).

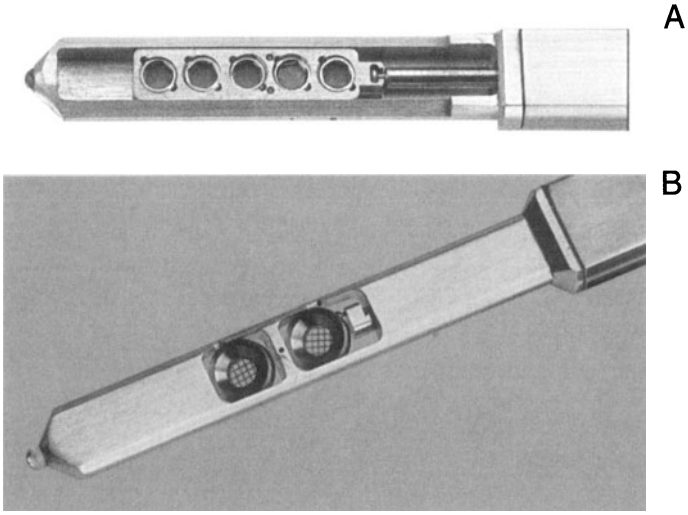

Figure 8.9. Multiple-specimen holders. (A) Five-specimen single-tilt and (B) two-specimen double-tilt.

So don't always think that you are limited to 3-mm specimens!

- *Double-tilt holder:* This is the most popular holder since it gives you the most flexibility in orienting the specimen. It is absolutely essential for imaging and diffraction studies of crystalline specimens. The tilt axes are fixed as two orthogonal directions. In some designs, you can

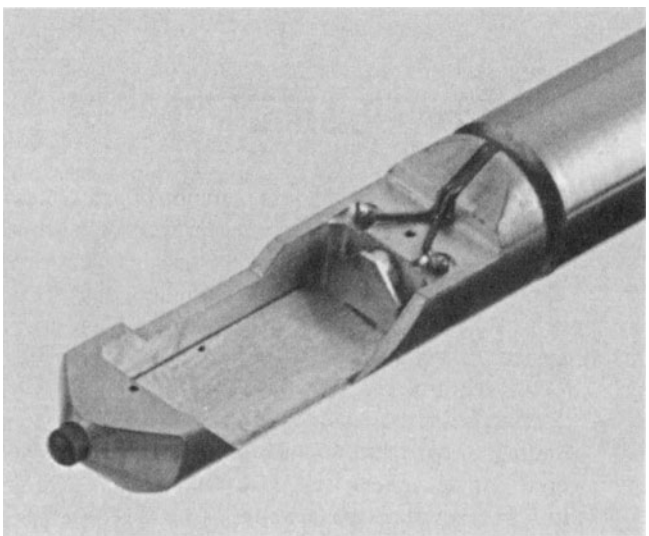

Figure 8.10. A bulk holder for large specimens.

remove the cup while the specimen is in place, which means that you can reinsert your specimen in the same orientation. This feature is extremely useful if your specimen is robust.

- *Tilt-rotate holder:* You would often like to be able to select the tilt axis. This holder lets you do just that and is particularly advantageous for the side-entry holder, since the tilt axis is then always parallel to the rod of the holder which also gives the largest tilt angle.
- *Low-background holder:* The cup and clamping ring are made of Be to minimize the generation of bremsstrahlung X-rays and characteristic X-rays. So they are required for XEDS studies. They can be double or single tilt and may be cooled also.
- *Heating holder:* Such holders in a conventional TEM can go to ~1300°C, which is measured by a thermocouple attached to the cup. In HVEMs, the temperature can go higher because of the larger gap between the polepieces. You have to be careful to calibrate the temperature and remember that the temperature may be different for different specimens. You should also be sure that the material you are studying does not form a eutectic alloy with the material forming the holder! If the eutectic does form, it will have a lower melting point, so you may deposit part of your specimen and the holder on the objective lens, or down onto the screen, if the microscope is well aligned.
- *Cooling holder:* This is available for either liquid-N_2 or liquid-He temperatures. These holders, which can be single or double tilt, are a great asset for XEDS, EELS, and CBED studies since they minimize surface-borne contamination. They are also essential for *in situ* studies of superconducting materials (both high and low T_c) and ideal for polymers or biological tissue. However, you should remember that the cold holder can also act as a small cryopump, so that it actually attracts contamination. Since you are necessarily changing the temperature at the specimen relative to its surroundings, be prepared for specimen drift. It takes time for the whole system to stabilize.
- *Cryo-transfer holder:* Certain specimens are prepared at cryogenic temperatures such as liquids, latex emulsions, and tissue in general. This holder permits you to transfer such cold specimens into the TEM without water vapor from the atmosphere condensing as ice on the surface.
- *Straining holder:* This holder clamps the specimen at both ends and then applies a load to one end, via a load cell or screw-thread mechanism, as shown in Figure 8.11. The sample is often in the shape of a small tensile specimen and is thinned in the middle of the gauge length (see inset). The motion of dislocations, cracks, etc. are then easily monitored, so a video camera is an essential accessory. You can vary the load to study cyclic as well as tensile loading, and the strain rate is another variable that is easily controlled. In Figure 8.11 a furnace is present, so the specimen can be heated while under load.
- *EBIC and CL holders:* The essential feature is the electrical feed-through that allows you to control the charge recombination in a semiconductor or certain mineral specimens by applying a bias across the specimen surface.

Beware: Heating and straining holders, in particular, can produce effects in thin foils that are totally uncharacteristic of your bulk specimen. So you must use these holders carefully and interpret your results cautiously. Often, surface reactions will dominate internal reactions when you are trying to induce a phase transformation by heating. The surface may also stop grain boundaries from migrating at temperatures where they would do so in the bulk material. Obviously, defect motion under applied stress may also be strongly affected since the 3D stress

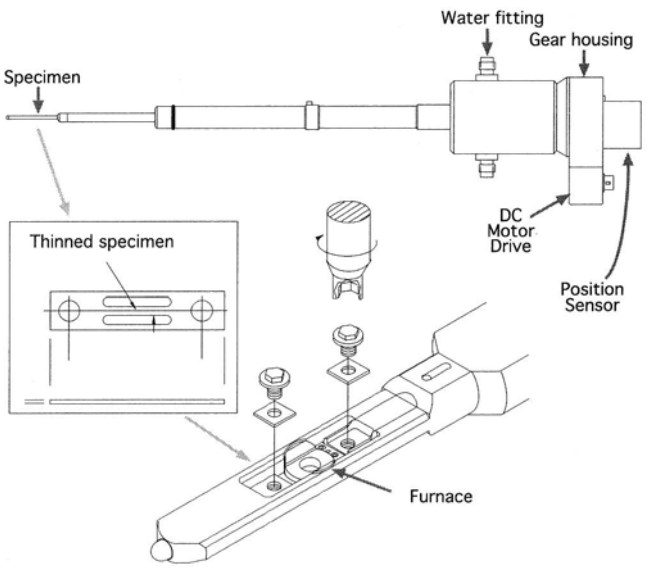

Figure 8.11. A side-entry combined straining and heating holder. The specimen looks like a miniature tensile specimen (inset) and is clamped at either end by hex screws. There is a screw-thread arrangement for pulling the specimen contained within the rod. The furnace surrounds the central thin portion of the specimen.

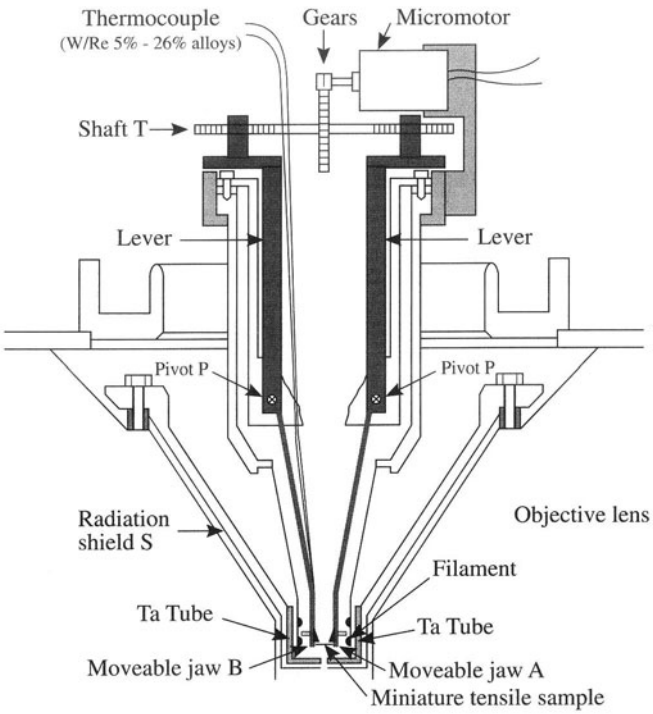

Figure 8.12. A top-entry, heating-straining holder which can be used at temperatures up to 2300 K in a 3-MV HVEM.

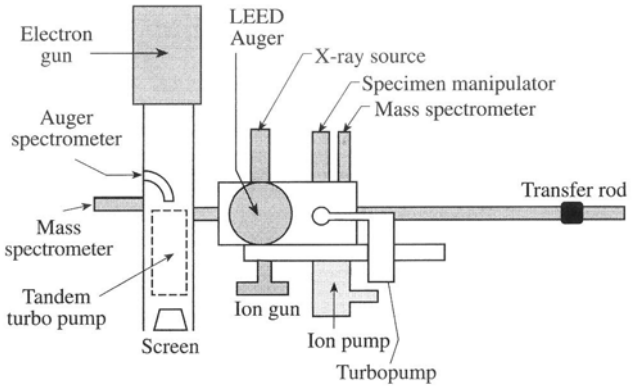

Figure 8.13. Schematic diagram showing the Hitachi H9000 UHV TEM. This instrument is equipped with a prechamber with LEED, Auger, and an ion gun which can be used to clean the specimen, allowing UHV surface analysis to be carried out on the TEM specimen. The holder has to transfer the specimen through a prepump chamber where it is ion-cleaned before going into the column.

field will be very different in bulk specimens compared to thin foils.

These problems can be overcome to some extent if you use thicker specimens and examine them in an HVEM, or at least an IVEM, and the whole field of *in situ* studies, particularly heating and/or straining, is best performed in such microscopes (Butler and Hale 1981 and Section 31.12). However, the high-energy electrons in these microscopes may introduce lattice defects that affect the very phenomenon that you want to study, e.g., beam-induced vacancies can change diffusional phase transformation kinetics very easily.

It is also possible, but much more difficult and expensive, to manipulate specimens in top-entry stages. The top-entry holder shown in Figure 8.12 is a heating-straining holder which is reported to be capable of operating at temperatures up to 2300 K (Komatsu *et al.* 1994). The heat is provided by a coaxial Ta tube which supports the W heater filament, as shown in the figure. The holder is used in a 3-MV microscope where the specimen diameter is 5 mm. The larger specimen diameter means that the disk can be shaped as a small tensile specimen and still be quite robust.

There are also special combinations of holders and stages which have been optimized for particular applications. The example shown in Figure 8.13 has been optimized to combine surface studies using low-energy electron diffraction (LEED) and Auger analysis with TEM. The prechamber is fitted with an ion gun to clean the sample before the surface is analyzed. The specimen can then be moved on into the TEM column for transmission studies. A similar prechamber has been used elsewhere to provide a method to clean the sample before growing thin films on the surface by molecular-beam epitaxy (MBE) or thermal evaporation.

One of the reasons for using higher accelerating voltages is that this gives more room in the specimen-stage region. Thus even 400-kV microscopes can be fitted with a small, differentially pumped environmental chamber. Such a chamber allows *in situ* studies of corrosion, degradation of catalysts, etc., especially when combined with a heating holder.

The article by Valdrè and Goringe (1971) gives a detailed description of several TEM holders.

CHAPTER SUMMARY

The vacuum and the holder are the two parts of the TEM that most closely affect your specimen. You have to treat both carefully if you want to be sure of getting the most out of your TEM. The vacuum is usually automated, so you don't have too much control over it. However, you can degrade the vacuum easily if you are a careless operator; for example, if you don't bother to prepump your film, and if you handle the specimen holder without gloves. In fact, you should treat the

specimen holder as if it were a rare jewel; it may actually contain a couple of synthetic ones and it certainly costs as much as a diamond of several carats!

With the range of holders available today, you can conduct many standard materials science experiments on your thin specimen while observing it in the TEM. However, if you're looking at crystalline material, the most common manipulation remains tilting in two orthogonal directions to orient different crystal planes parallel to the electron beam. You'll understand why this is important after you've finished reading Parts II and III.

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